

**STUDY ON THE EFFECT OF HIGH ENERGY BALL  
MILLING (A NANO MATERIAL PROCESS) ON THE  
MICROSTRUCTURE AND MECHANICAL PROPERTIES  
OF A (Al-Si-Fe-Cu) ALLOY**

A THESIS SUBMITTED IN PARTIAL FULFILLMENT  
OF THE REQUIREMENTS FOR THE DEGREE OF

**Bachelor of Technology  
In  
Metallurgical and Materials Engineering**

By  
**KAUSHAL KISHORE SINGH  
SUDIPTO BHATTACHARJEE**



**Department of Metallurgical and Materials Engineering  
National Institute of Technology  
Rourkela  
2007**

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Under the Guidance of  
**Prof. Dr.A.K.PANDA**



**Department of Metallurgical and Materials Engineering**  
**National Institute of Technology**  
**Rourkela**  
2007



**National Institute of Technology  
Rourkela**

**CERTIFICATE**

This is to certify that the thesis entitled, “Study on the effect of high energy ball milling (a nano material process) on the microstructure and mechanical properties of a(Al-Si-Fe-Cu) alloy” submitted by Sri Kaushal Kishore Singh and Sri Sudipto Bhattacharjee in partial fulfillment of the requirements for the award of Bachelor of Technology Degree in Metallurgical and Materials Engineering at the National Institute of Technology, Rourkela (Deemed University) is an authentic work carried out by them under my supervision and guidance.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University / Institute for the award of any Degree or Diploma.

Date:

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April, 2007

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**ABSTRACT**

Mechanical alloying is a solid-state powder processing technique involving repeated welding, fracturing, and rewelding of powder particles in a high-energy ball mill. Mechanical Alloying has now been shown to be capable of synthesizing a variety of equilibrium and non-equilibrium alloy phases starting from blended elemental or prealloyed powders. Materials have been found to attain unusual properties in their nano level sizes which are not found in their conventional form. Al-Si alloys containing other transition metals are used in the as cast condition for automotive and aerospace for the engine components.

Srivastava et al have studied Al-18%Si-5%Fe-1.5%Cu by rapid solidification technique and have compared the microstructure with that in the cast condition. In the present work an attempt was made to develop this alloy through mechanical alloying route.

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# **Chapter 1**

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## **INTRODUCTION**

# INTRODUCTION

Nanomaterials are experiencing a rapid development in recent years due to their existing and/or potential applications in a wide variety of technological areas such as electronics, catalysis, ceramics, magnetic data storage, structural components etc. To meet the technological demands in these areas, the size of the materials should be reduced to the nanometer scale. For example, the miniaturization of functional electronic devices demands the placement or assembly of nanometer scale components into well-defined structures. As the size reduces into the nanometer range, the materials exhibit peculiar and interesting mechanical and physical properties, e.g. increased mechanical strength, enhanced diffusivity, higher specific heat and electrical resistivity compared to conventional coarse grained counterparts. Nanomaterials (nanocrystalline materials) are materials possessing grain sizes on the order of a billionth of a meter. They manifest extremely fascinating and useful properties, which can be exploited for a variety of structural and non-structural applications. Nanomaterials can be classified into nanocrystalline materials and nanoparticles. The former are polycrystalline bulk materials with grain sizes in the nanometer range (less than 100 nm), while the latter refers to ultra fine dispersive particles with diameters below 100 nm. Nanoparticles are generally considered as the building blocks of bulk nanocrystalline materials. Research in nanomaterials is a multidisciplinary effort that involves interaction between researchers in the field of physics, chemistry, mechanics and materials science, or even biology and medicine. It has been stimulated by the interest for basic scientific investigations and their technological applications. Nanomaterials and most of the applications derived from them are still in an early stage of technical development. There are several issues that remain to be addressed before nanomaterials will become potentially useful for industrial sectors. These issues include synthesis of high purity materials with large yield economically and environmentally, characterization of new structures and properties of nanophase materials, fabrication of dense products from nanoparticles with full density and less contamination, and retention of the ultrafine grain size in service in order to preserve the mechanical properties associated with the nanometer scale. Novel fabrication technology of nanoparticles is versatile and includes a wide range of vapor, liquid and solid state processing routes.

Available techniques for the synthesis of nanoparticles via vapor routes range from physical vapor deposition and chemical vapor deposition to aerosol spraying. The liquid route involves sol-gel and wet chemical methods. The solid-state route preparation takes place via mechanical milling and mechanochemical synthesis. Each method has its own advantages and shortcomings. Among these, mechanical milling and spray conversion processing are commonly used to produce large quantities of nanopowders. Nanoparticles synthesized from several routes may have different internal structures that would affect the properties of materials consolidated from them. Processing nanoparticles into fully dense, bulk products or coatings which retain the nanometer scale grain size is rather difficult to achieve in practice. Due to their high specific surface areas, nanoparticles exhibit a high reactivity and strong tendency towards agglomeration. Moreover, rapid grain growth is likely to occur during processing at high temperatures. As unique properties of nanocrystalline materials derived from their fine grain size, it is of crucial importance to retain the microstructure at a nanometer scale during consolidation to form bulk materials. It is also noticed that pores are generated in bulk nanocrystalline materials consolidated from nanoparticles prepared by inert-gas condensation. Such nanopores can lead to a decrease in Young's modulus of consolidated nanocrystalline materials. Electrodeposited samples are believed to be free from porosity, but they contain certain impurities and texture that may degrade their mechanical performances. Therefore, controlling these properties during synthesis and subsequent consolidation procedures are the largest challenges facing researchers. The unique properties of nanocrystalline materials are derived from their large number of grain boundaries compared to coarse-grained polycrystalline counterparts. In nanocrystalline solids, a large fraction of atoms (up to 49%) are boundary atoms. Thus the interface structure plays an important role in determining the physical and mechanical properties of nanocrystalline materials. Huang reported that nanocrystalline copper has a much higher resistivity and a larger temperature dependence of the resistivity than bulk copper. They attributed this effect to the grain boundary enhanced scattering of electrons. Despite recent advances in the development of nanocrystalline materials, much work remains to be done to achieve a basic understanding of their deformation and fracture behaviour.

## 1.1 Properties of nanomaterials

Nanostructured materials are a broad class of materials, with microstructures modulated in zero to three dimensions on length scales less than 100 nm. These materials are atoms arranged in nanosized clusters, which become the constituent grains or building blocks of the material. Conventional materials have grains sizes ranging from microns to several millimetres and contain several billion atoms each. Nanometre sized grains contain only about 900 atoms each. As the grain size decreases, there is a significant increase in the volume fraction of grain boundaries or interfaces. This characteristic strongly influences the chemical and physical properties of the material.

Nanophase metals exhibit significant increases in yield strength and elastic modulus. It has also been shown that other considerable interest in the generation of carbon nanostructures, which are related to the famous Buckyball. In addition, the use of nano-sized materials as fillers for composite materials is generating interest. Specifically in the case of polymer nanocomposites. All materials are composed of grains, which in turn comprise many atoms. These grains are usually invisible to the naked eye, depending on their size. Conventional materials have grains varying in size anywhere from 100's of microns ( $\mu\text{m}$ ) to millimetres (mm). A nanometre (nm) is even smaller a dimension than a  $\mu\text{m}$ , and is a billionth ( $10^{-9}$ ) of a meter. A nanocrystalline material has grains on the order of 1-100 nm. The average size of an atom is on the order of 1 to 2 angstroms ( $\text{\AA}$ ) in radius. 1 nanometre comprises 10  $\text{\AA}$ , and hence in one nm, there may be 3-5 atoms, depending on the atomic radii. Nanocrystalline materials are exceptionally strong, hard, and ductile at high temperatures, wear-resistant, erosion-resistant, corrosion-resistant, and chemically very active. Nanocrystalline materials, or nanomaterials, are also much more formable than their conventional, commercially available counterparts.

## **1.2 Method of synthesis**

There are five widely known methods of production of nanomaterials they are given as follows:

- ☐ Sol-gel synthesis.
- ☐ Inert gas condensation.
- ☐ Mechanical alloying or high-energy ball milling.
- ☐ Plasma synthesis.
- ☐ Electrodeposition.

### **1.2.1 Sol-Gel Synthesis:**

Sol-gel Synthesis is the synthesis of primarily metal oxides (binary, ternary...) by hydrolysis and condensation reactions of suitable precursors that has also been applied to non-oxide material.

### **12.2 Inert Gas Condensation:**

A modification that consists of a scraper and a collection funnel allows the production of relatively large quantities of nanoparticles, which are agglomerated but do not form hard agglomerates and which can be compacted in the apparatus itself without exposing them to air. The group of Birringer and Gleiter pioneered this method. Increased pressure or increased molecular weight of the inert gas leads to an increase in the mean particle size. This so-called Inert Gas Condensation method is already used on a commercial scale for a wide range of materials.

### **1.2.3 Mechanical Alloying or High Energy Milling:**

Mechanical alloying was originally invented as a method to manufacture oxide dispersion strengthened nickel alloys. It is a high energy ball milling process, where alloying is the result of the repeated fracture and cold welding of the component particles. Highly metastable materials such as amorphous alloys and nanostructured materials can be prepared by the process. Scaling up to industrial quantities seems straight forward. In addition to attrition and agglomeration, high energy milling can induce chemical reactions, which can be used to influence the milling process and the properties of the product. This fact was utilized to prepare magnetic oxide-metal nanocomposites via mechanically induced displacement reactions between a metal oxide and a more reactive metal. High energy ball milling can also induce chemical changes in non-metallurgical binary systems, including silicates, minerals, ferrites, ceramics, and organic compounds. The research area of mechanochemistry was developed to study and utilize these processes. As many mechanical alloying processes involve chemical changes, the distinction between mechanical alloying and mechanochemistry is often arbitrary.

### **1.2.4 Plasma Synthesis:**

Plasma Synthesis has been used in the preparation of advanced materials such as new ceramics, nanometric metallic powders, biomaterials and superconductors. The typical size of the nanoparticles produced ranges from 20 to 100 nm, depending on the quench conditions employed. Such small-scale materials display unusual properties (chemical, physical, electrical, optical, mechanical, magnetic, etc.) as compared to their bulk states. The production of nanopowders is one of the most notable applications of induction plasma. The induction plasma torch possesses a high flexibility and tolerance to the processing chemistry because there are no metallic electrodes to react with the reactants, and therefore oxidising or reducing atmospheres can be used.

### **1.2.5 Electrodeposition:**

Electrodeposition is an electrode reactions involving oxidation/reduction of a solid metal and its dissolved ion. e.g., if a copper metal rod is immersed in a copper sulphate solution, the copper cations can be cathodically reduced to copper metal, or the copper metal can be anodically oxidized to copper ions. Compare with a redox reaction where both the oxidized and the reduced species are in solution. The terms "Electrodeposition" and "Electrodissolution" are often used to describe these reactions.

### **1.2.6 Epitaxy:**

This technology is quite similar to what happens in CVD processes, however, if the substrate is an ordered semiconductor crystal (i.e. silicon, gallium arsenide), it is possible with this process to continue building on the substrate with the same crystallographic orientation with the substrate acting as a seed for the deposition. If an amorphous/polycrystalline substrate surface is used, the film will also be amorphous or polycrystalline.

# **Chapter 2**

**LITRATURE SURVEY**



## **LITRATURE SURVEY**

### **2.1APPLICATION OF NANO MATERIALS**

Since nanomaterials possess unique, beneficial chemical, physical, and mechanical properties, they can be used for a wide variety of applications. These applications include, but are not limited to, the following:

- High strength application.
- Electric and magnetic material.
- High-sensitivity sensors.
- Hydrogen storage.
- Biotechnology.
- Semi conducting properties of dielectric.
- Kinetic Energy (KE) penetrators with enhanced lethality.
- High energy density batteries.
- Longer-lasting satellites.
- Longer-lasting medical implants.
- Large electrochromic display devices.

## 2.2 THERMODYNAMICS OF SOLUTION FORMATION

The alloy is in a mechanically homogenized nonequilibrium state, and that prolonged annealing at low temperatures should lead to, for example, the clustering of chromium atoms. Solution formation normal thermodynamic theory for solutions begins with the mixing of component atoms. In mechanical alloying, however, the solution is prepared by first mixing together lumps of the components, each of which might contain many millions of identical atoms. We examine here the way in which a solution evolves from these large lumps into an intimate mixture of different kinds of atoms without the participation of diffusion or of melting and this leads to interesting outcomes which have implications on how we interpret the mechanical alloying process. Consider the pure components A and B with molar free energies  $\mu_A^0$  and  $\mu_B^0$  respectively. If the components are initially in the form of powders then the average free energy of such a mixture of powders is simply:

$$G\{\text{mixture}\} = (1-x)\mu_A^0 + x\mu_B^0 \quad ; \text{ where } x \text{ is the mole fraction of B}$$

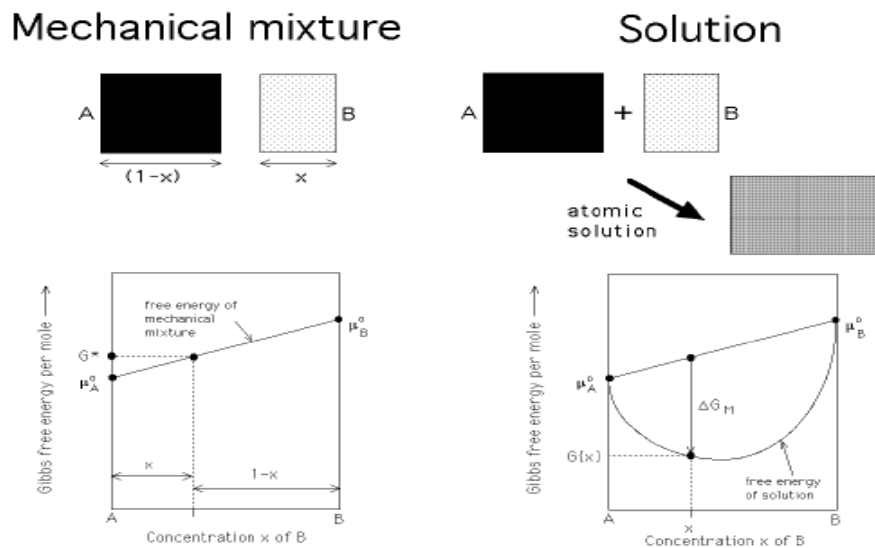


Fig. 2.1: (a) The free energy of a mechanical mixture, where the mean free energy is simply the weighted mean of the components. (b) The free energy of an ideal atomic solution is always lower than that of a mechanical mixture due to configurational entropy.

It is assumed that the powder particles are so large that the A and B atoms do not “feel” each other’s presence via interatomic forces between unlike atoms. It is also assumed that the number of ways in which the mixture of powder particles can be arranged is not sufficiently different from unity to give a significant contribution to configurational entropy of mixing. It has a free energy that is simply a weighted mean of the components. In contrast to a mechanical mixture, a solution is conventionally taken to describe a mixture of atoms or molecules.

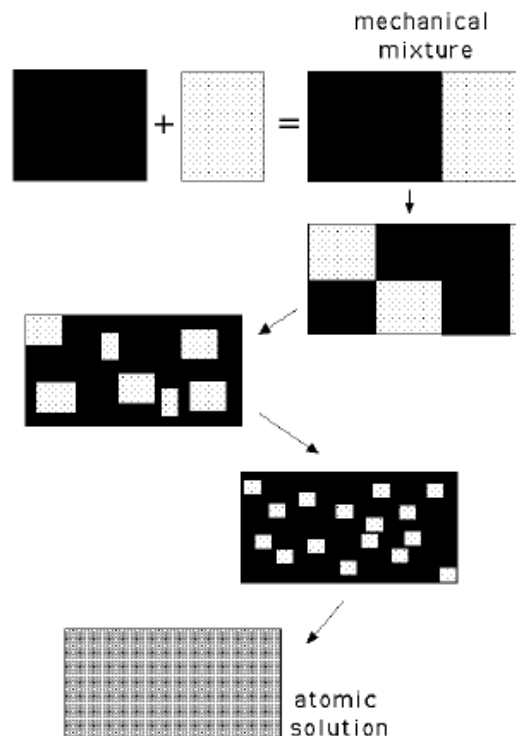


Fig. 2.2: Schematic illustration of the evolution of an atomic solution by the progressive reduction in the size of different particles, a process akin to mechanical alloying.

There will in general be an enthalpy change associated with the change in near neighbour bonds. Because the total number of ways in which the “particles” can arrange is now very large, there will always be a significant contribution from the entropy of mixing, even when the enthalpy of mixing is zero. The free energy of the solution is therefore different from that of the mechanical mixture. The difference in the free energy between these two states of the components is the free energy of mixing, the. The problem is illustrated in Fig.2.2 which shows the division of particles into ever smaller particles until an atomic solution is achieved.

### 2.3 TYPICAL CHEMICAL COMPOSITION

The intense deformation associated with mechanical alloying can force atoms into positions where they may not prefer to be at equilibrium. The atomic structure of solid solutions in commercially important metals formed by mechanical alloying process can be studied using field ion microscopy and atom probe.

Fe-base	C	Cr	Al	Mo	Ti	N		Ti <sub>2</sub> O <sub>3</sub>	Y <sub>2</sub> O <sub>3</sub>	Fe
MA956	0.01	20.0	4.5	–	0.5	0.045		–	0.50	Balance
PM2000	< 0.04	20.0	5.5		0.5			–	0.5	Balance
Ni-Base	C	Cr	Al	Ti	W	Fe	N	Total O	Y <sub>2</sub> O <sub>3</sub>	Ni
MA6000	0.06	15.0	4.5	2.3	3.9	1.5	0.2	0.57	1.1	Balance
PM1000 †		20.0	0.3	0.5		3.0			0.6	Balance

Table.2.1: Compositions (wt %) of some typical alloys.

## 2.4 PROCESS VARIABLES

Mechanical alloying is a complex process and hence involves optimization of a number of variables to achieve the desired product phase and/or microstructure. Some of the important parameters that have an effect on the final constitution of the powder are:

- Type of mill
- Milling container
- Milling speed
- Milling time
- Type, size, and size distribution of the grinding medium
- Ball-to-powder weight ratio
- Extent of filling the vial
- Milling atmosphere
- Process control agent
- Temperature of milling.

All these process variables are not completely independent. For example, the optimum milling time depends on the type of mill, size of the grinding medium, temperature of milling, ball-to-powder ratio, etc

## **2.5 TYPE OF MILLING EQUIPMENTS USED FOR MECHANICAL ALLOYING**

Different types of high-energy milling equipment are used to produce mechanically alloyed powders. They differ in their capacity, efficiency of milling and additional arrangements for cooling, heating, etc.

- SPEX shaker mills
- Attritor mills
- Commercial mills
- Planetary ball mills (To be used in our experimentation)

### **2.5.1 SPEX shaker mills**

Shaker mills such as SPEX mills, which mill about  $10 \pm 20$  g of the powder at a time, are most commonly used for laboratory investigations and for alloy screening purposes.



Fig-2.3: SPEX shaker mill

### 2.5.2 Attritor mills

A conventional ball mill consists of a rotating horizontal drum half-filled with small steel balls. As the drum rotates the balls drop on the metal powder that is being ground.

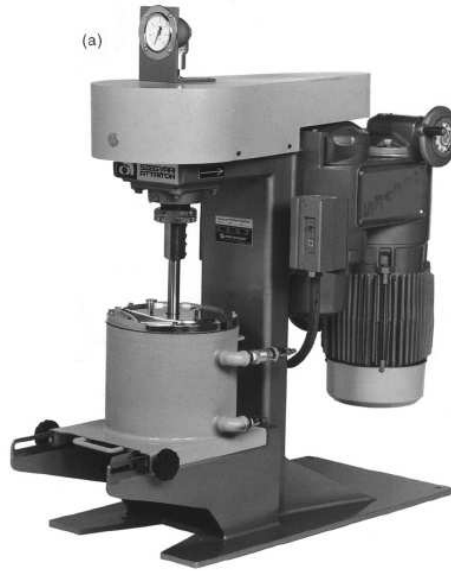


Fig 2.4: Attritor mill

### 2.5.3 Planetary ball mills:

Another popular mill for conducting MA experiments is the planetary ball mill (referred to as Pulverisette) in which a few hundred grams of the powder can be milled at a time. The planetary ball mill owes its name to the planet-like movement of its vials. These are arranged on a rotating support disk and a special drive mechanism causes them to rotate around their own axes. The centrifugal force produced by the vials rotating around their own axes and that produced by the rotating support disk both act on the vial contents, consisting of material to be ground and the grinding balls. Since the vials and the supporting disk rotate in opposite directions, the centrifugal forces alternately act in like and opposite directions. This causes the grinding balls to run down the inside wall of the vial the friction effect, followed by the material being ground and grinding balls lifting off and traveling freely through the inner chamber of the vial and colliding against the opposing inside wall the impact effect (Fig.2.6).



Fig.2.5: Planetary ball mill

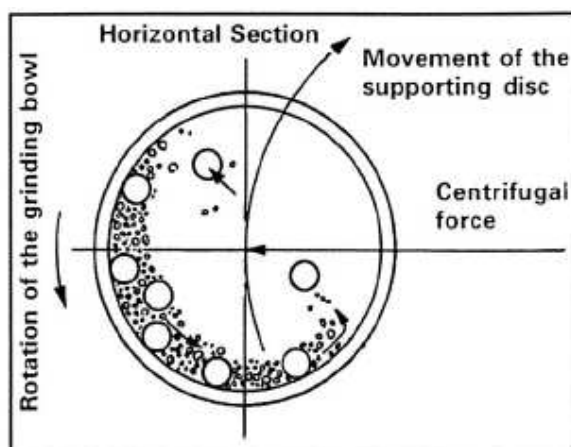


Fig-2.6: Showing types of forces acting

Even though the disk and the vial rotation speeds could not be independently controlled in the earlier versions, it is possible to do so in the modern versions. Grinding vials and balls are available in eight different materials agate, silicon nitride, sintered corundum, zirconia, chrome steel, Cr+Ni steel, tungsten carbide, and plastic polyamide. Even



though the linear velocity of the balls in this type of mill is higher than that in the SPEX mills, the frequency of impacts is much more in the SPEX mills.

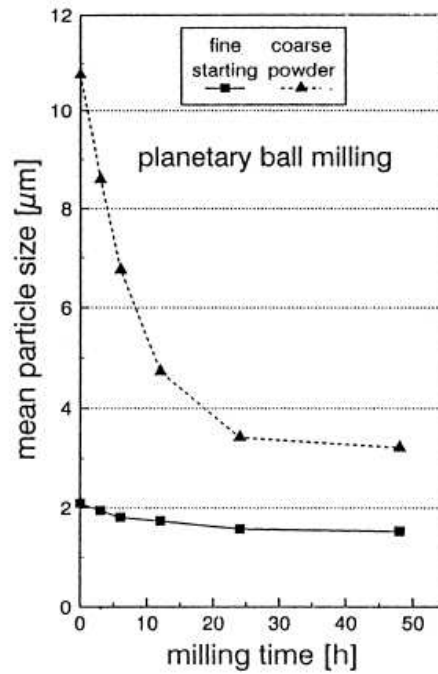


Fig-2.7: Plot of Particle size Vs Milling time

## 2.7 MECHANISM OF MECHANICAL ALLOYING

- In the planetary ball mill its vials are arranged on a rotating support disk.
- A special drive mechanism causes them to rotate around their own axes.
- The force of the impact plastically deforms the powder particles.
- The particles get work hardened and fracture.
- The new surfaces created enable the particles to weld together.
- Particle contains substantially all of the starting ingredients, in the proportion they were mixed together

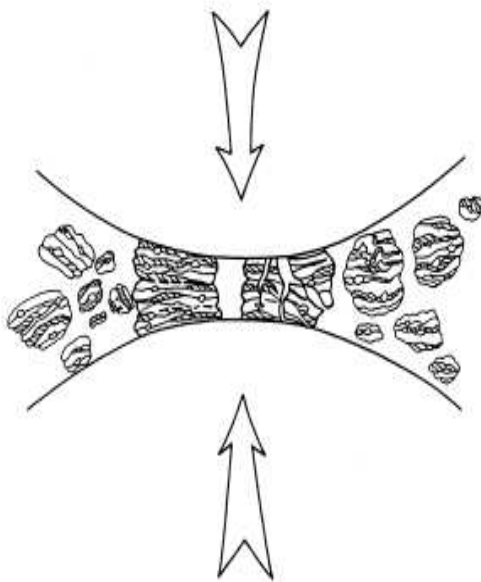


Fig-2.8: Ball-powder-ball collision of powder mixture during mechanical alloying.

## 2.8 PREVIOUS WORK DONE

1. Development of magnetic and superconducting properties.eg: Oxide superconductor:  $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$  by plasma synthesis processing of fine particles, especially of high-tech ceramic materials. Peter C. Kong and Y. C. Lau, Pure & App/. Chem., Vol. 62, No. 9, pp. 1809-1816, 1990,
2. Aluminium alloy with nominal composition of Al–18%Si–5%Fe–1.5%Cu (wt.%) has been spray deposited and its microstructural features have been studied V.C.Srivastava , S.N.Ojha, P.Ghosal. Materials Letters vol 56 (2002) ,page797– 801.
3. Lithium ion intercalation is done to enhance its anodic properties. Ball-milling in liquid media. Raphafel Janot, Daniel Guerard , Materials Science 50 (2005) 1–92
4. Nanocrystalline materials and coatings. S.C. Tjong, Haydn Chen. Materials Science and Engineering R 45 (2004) 1–88.
5. Concerning the magnetic properties, some fine-particle systems exhibit good hard magnetic properties and used as soft magnets R. Hamzaoui, O. Elkedim, N. Fenineche, E. Gaffet, J. Craven Materials Science and Engineering A360 (2003) 299- 305.

# **Chapter 3**

**EXPERIMENTAL**

## EXPERIMENTAL

The composition which has been studied:

Al-18%Si-5%Fe-1.5%Cu

Stoichiometric amounts of the powders are weighed and mixed. Milling carried out in a planetary ball mill. In Planetary ball mill the milling was schedule as milling time 10 min and pause 15min, this was done because in milling high amount of energy is evolved and it can cause hazards. The milling was carried out in the medium, in order to maintain the powder mixture at at room temperature. Type of container used was  $\text{ZrO}_2$ . Total milling time employed was 25Hrs with suitable milling speed of 300rpm.

### 3.1X-RAY DIFFRACTION

Samples were taken out for XRD analysis after for every 5 Hrs of milling which was carried out in Phillips expert system. Fig-3.1 shows the typical diffractograms. The comparison of the diffractograms reveals progressing line broadening as a function of time of milling, Fig- shows the overlapped first peak of Aluminium.

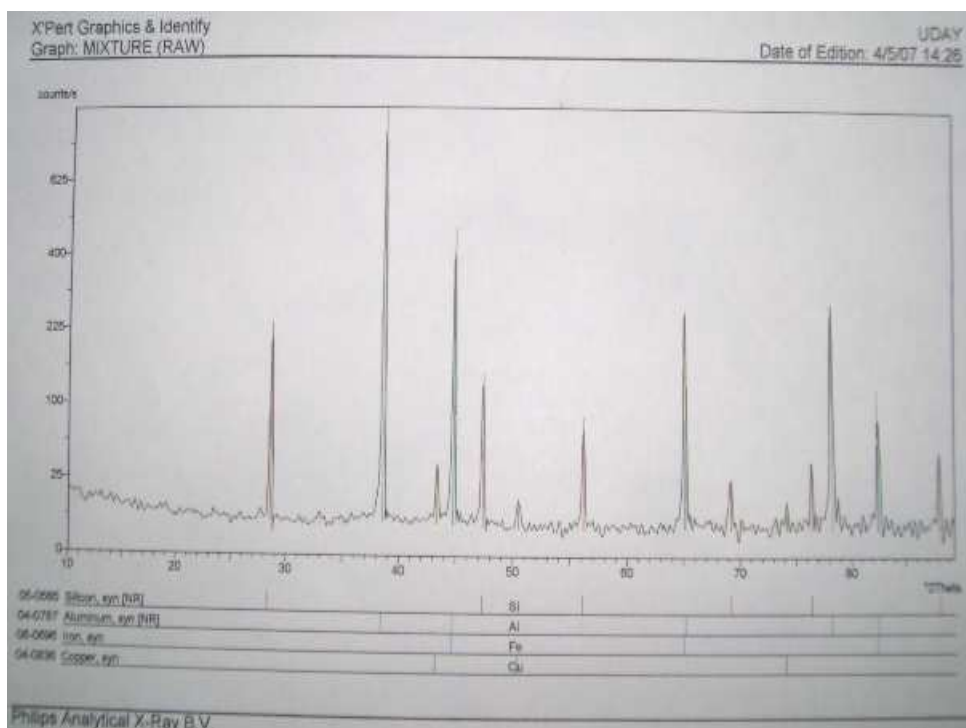


Fig-3.1: Typical X-ray diffractogram of mechanically alloyed (Al-18%Si-5%Fe-1.5%Cu) powder.

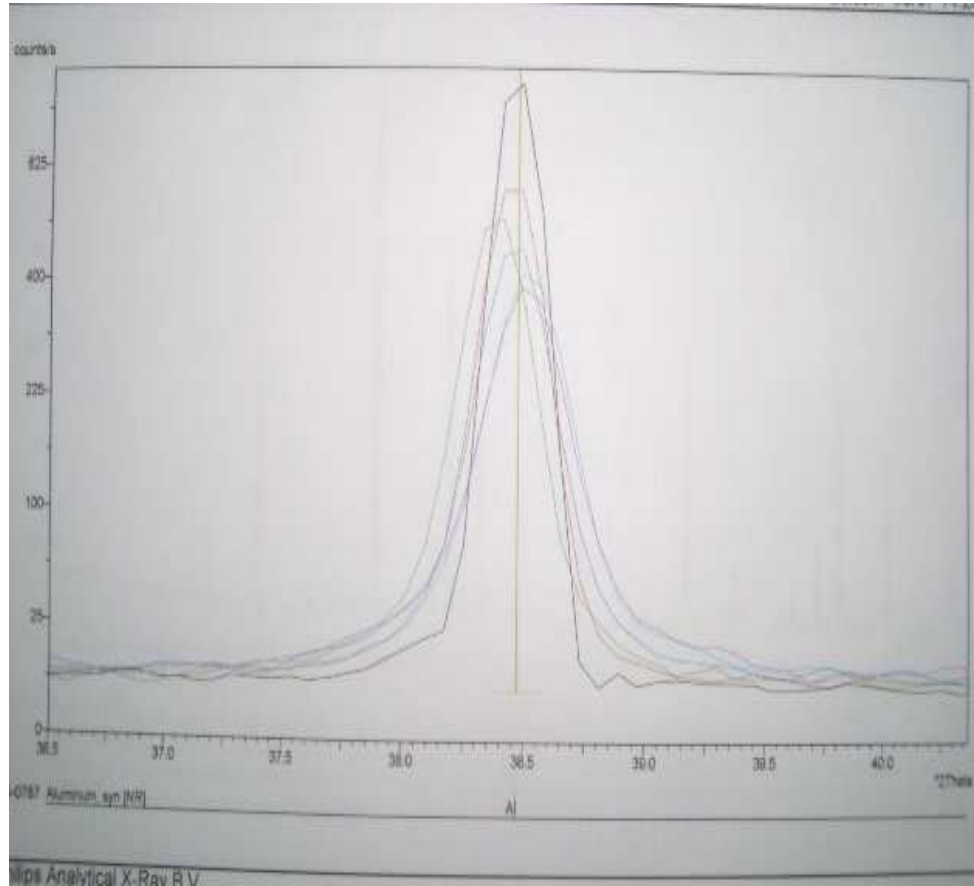


Fig-3.2 Shows the overlapped first peak of Aluminium

Fig-3.2 shows the comparison between the different diffractograms for the first peak of aluminium for the samples obtained by simple mixing and milled for 5,10,15,20,25 hrs and it reveals progressing line broadening as a function of time of milling.

### 3.2 PARTICLE SIZE CALCULATION

The crystallite size and lattice strain in the powder particles can be determined by the X-ray peak broadening techniques. X-ray diffraction peaks are broadened due to:

1. Instrumental effects,
2. Small particle size,
3. Lattice strain in the material.

However, if one is interested only in following the trend of change of crystallite size with milling conditions, this simple technique may be acceptable. While the X-ray peak broadening due to small crystallite size is inversely proportional to  $\cos \theta$ . The decrease in particle size with increase can be seen in Table 3.1

Table-3.1

S/No.	Milling Time (Hrs)	Particle Size(Angstrom)
1	5	283.83
2	10	266.96
3	15	256.81
4	20	237.95
5	25	238.22

Most commonly the crystallite size is determined by measuring the Bragg peak width at half the maximum intensity and putting its value in Scherrer's formula which is mentioned below:

**Scherrer formula:**

$$d=0.9 \lambda / (B \cos\theta)$$

Where:

d is the crystallite size,

$\lambda$  is the wavelength of the X-radiation used,

B is the peak width at half the maximum intensity, and  $\theta$  is the Bragg angle.

The particle size as a function of milling time is shown in Table-3.1 and the curve is plotted and shown in Fig-3.3

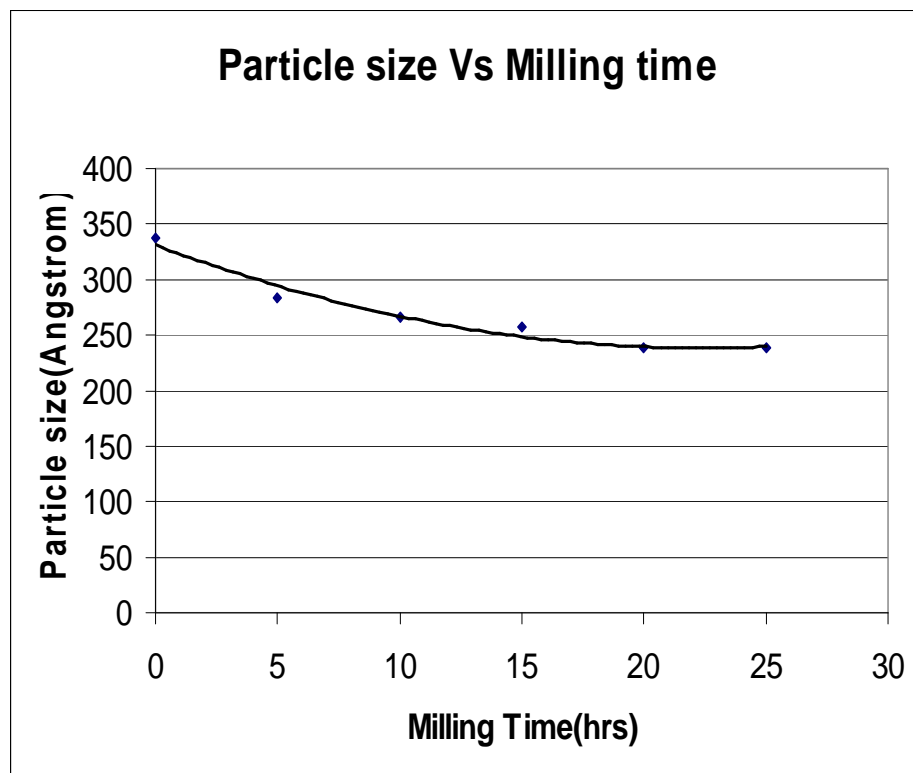


Fig-3.3: Particle size as a function of milling time.



# **Chapter 4**

## **RESULTS AND DISCUSSION**

## RESULTS AND DISCUSSION

XRD analysis shows a compound formation between Al,Fe,Si the exact compound is to be conformed .V.C. Srivastava, P. Ghosal, S.N. Ojha .has found compound between these element as  $\delta\text{Al}_4\text{Si}_2\text{Fe}$  and  $\beta\text{Al}_5\text{SiFe}$  using energy dispersive Xray analysis.The plot between particle size Vs milling time Fig- 3.3 reveals that particle has become finer with the increase in milling time.The milling has been carried out for 25 hrs and results are presented.Out of the mechanically alloyed powder mixture , green pellets of 10 & 15 mm dia.and 12mm height,by applying pressure of 6.5 tons using hydraulic machine.Then they were sintered at 450°C for ½ hr.

Additionally another set of experiment was planned which is as given below. Powder mixture of same composition was made using mixing machine resulting in ordinary mixing.This powder mixture was used for making green pellets followed by sintering adopting the schedule as mentioned in Table No.4.1

Table-4.1

SlNo.	Sintering temperature(°C) (X <sub>1</sub> )	Pressure(T) ( X <sub>2</sub> )
1	400	5
2	500	5
3	400	8
4	500	8
5	450	6.5

After green pellets were sintered one batch was tested in INSTRON 1195 for compressive strength where as the other batch was used for revealing the microstructure by optical microscopy. The compression strengths of the samples are tabulated along with the schedule of experiment and shown in Table-4.2

Table- 4.2

SINo.	Sintering temperature(oC) ( X1)	Compaction Pressure(Tons) (X2)	Compressive strength(MPa)
1	400	5	125.4
2	500	5	146
3	450	6.5	183.3
4	400	8	171.9
5	500	8	198.4

A regression equation was formulated by using the method of statistical design of experiments.

Compressive strength (MPa):  $160.425 + 11.77X_1 + 24.725X_2 + 1.475X_1X_2$

Where,  $X_1$  is the symbol assigned for sintering temperature and  $X_2$  for compaction pressure.

The coefficients of  $X_1$  and  $X_2$  have large positive values indicating that both temperature and pressure have large increasing influence ,though pressure has got larger influence than temperature .The experiment carried out with 450°C and 6.5 Ton compaction pressure resulted in a compressive strength of 183.3MPa which is quite close to the value of  $b_0$  is 160.425 MPa .Thus this equation is valid within the range of variables used. Table-4.3 shows a comparison between the compressive strength of the samples prepared by mixing machine and mechanical alloying.

Table-4.3

SlNo.	Type of mixing/milling	Sintering Temperature (°C)	Compaction Pressure(T)	Compressive Strength (MPa)
1.	Simple mixing	450	6.5	183.3
2.	Mechanical Alloying	450	6.5	212.5
3.	Mechanical Alloying	450	8	254.4

The compressive strength of the pellets made out of mechanically alloyed powder (time of milling of 25hrs and the compaction pressure applied was 6.5T & sintering temperature 450°C temperature and time of sintering ½ an hour) was found to be 212.5MPa. This value is higher than 183.3MPa obtained for the powder made out of ordinary mixing this value is attributed to the better bondage between the particles in mechanically alloyed sample because of high reactivity resulted out of creation of large surface .

Another mechanically alloyed sample but with 8tons of compaction pressure for same temperature 450°C mechanically alloyed but 8T of compaction temperature shows a compressive strength of 254.4MPa this is attributed to the more effective compaction and more effective bonding between particles.

## MICROSTRUCTURE

The SEM photographs of samples

- a. Mixed by simple mixing.
- b. Mechanically Alloyed (25 Hrs).

were taken after subjecting them to a compaction pressure of 6.5 Ton and a sintering temperature of 450°C for ½ hr. are shown in Fig-4.1 and Fig4.2

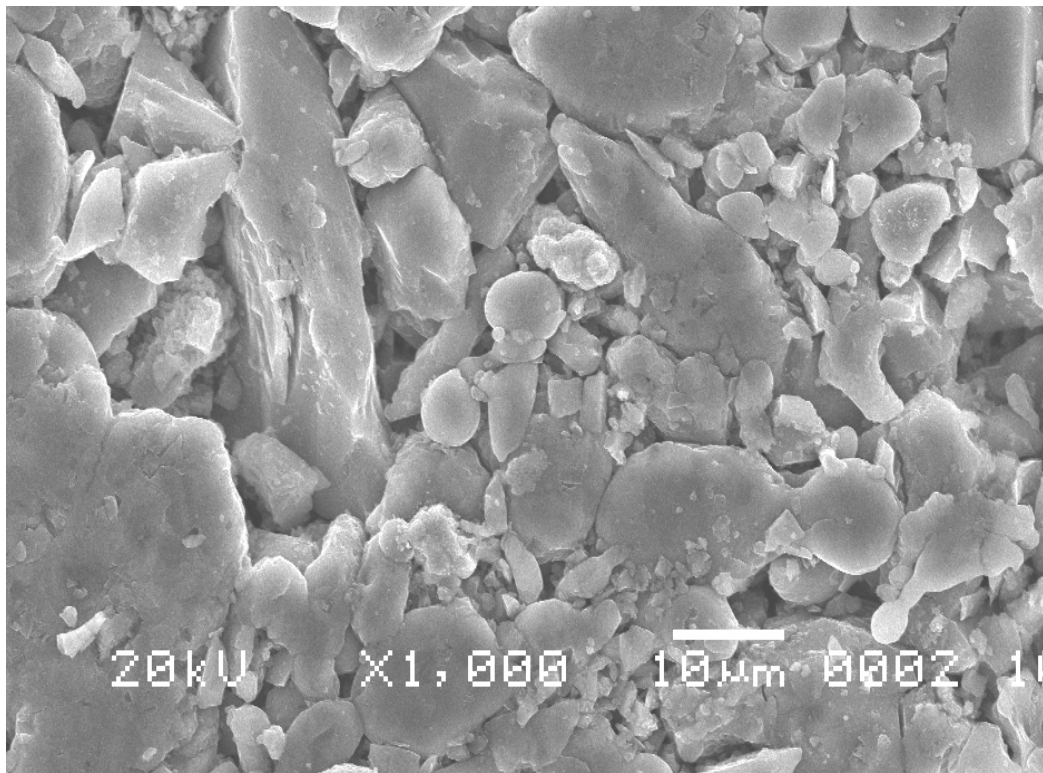


Fig-4.1: (1000X)

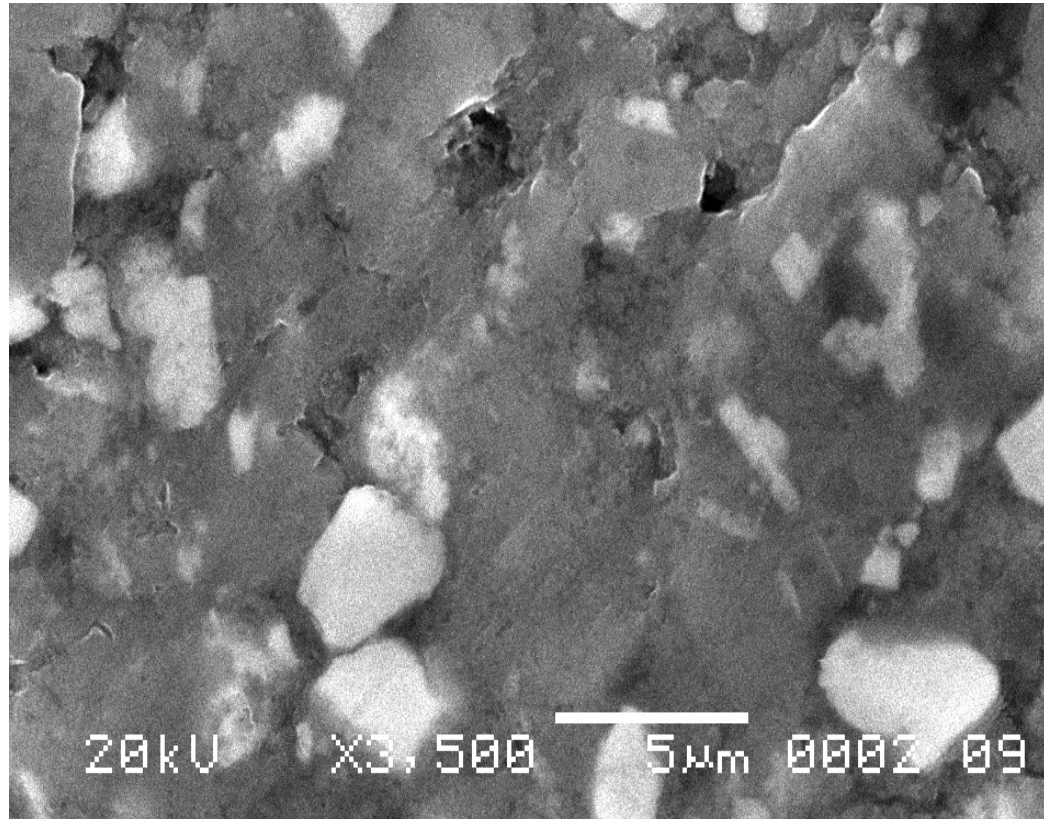


Fig-4.2: (3500X)

The microphotographs reveal the following features .In the ordinary mixed sample (6.5 T and 450°C) there are large number of pores (Fig.4.1), where as in the mechanically alloyed sample such pores are less in number (Fig.4.2).The size of the particles in a mechanically alloyed sample are comparable at 3500X(Fig.4.2) with those of ordinary mixed sample at 1000X(Fig.4.1).In addition grainboundaries can be seen in some areas in mechanically alloyed sample (Fig.4.2) indicating that alloy formation has occurred where as in ordinary mixed samples this is absent. There are some white phases presumably intermetallic compounds formed by Al-Fe-Si. in mechanically alloyed sample. This is revealed by XRD data analysis. The exact stoichiometric formula of this intermetallic compound between Al-Fe-Si is yet to be confirmed.

# Chapter 5

**CONCLUSION**

**REFERENCES**

## CONCLUSION

1. XRD shows a compound formation between Al, Fe, and Si. The exact composition is to be conformed.
2. The particles have become finer as a function of milling time as revealed by broadening of the XRD peaks.
3. Compaction pressure and sintering temperature has increasing influence, though pressure has got larger influence than temperature as revealed by the regression equation.



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